



Effect of bismuth telluride concentration on the thermoelectric properties of PEDOT:PSS–glycerol organic films



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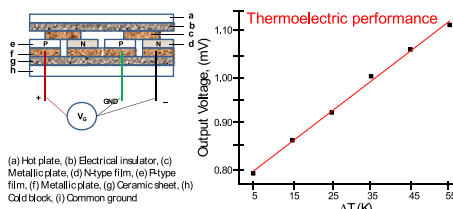
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HIGHLIGHTS

- Bismuth telluride (BT) doped PEDOT:PSS–Glycerol hybrid thin film exhibits promising thermoelectric properties.
- The hybrid film demonstrates tuneable thermoelectric properties via BT doping concentration adjustment.
- Thermoelectric device from p-type and n-type hybrid films indicated highly efficient for power generation.

GRAPHICAL ABSTRACT

Highly efficient thermoelectric device with output power as high as 6 W can be prepared using Bi₂Te₃ doped PEDOT:PSS–Glycerol hybrid films.



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ABSTRACT

In this work, the effect of bismuth–telluride concentration on the thermoelectric properties of PEDOT:PSS–Glycerol thin films is investigated. A thermoelectric device was fabricated by depositing the n-type and the p-type Bi₂Te₃ (BT) doped-PEDOT:PSS–Glycerol on a glass substrate via a spin coating method at 500 rpm. Room-temperature electrical properties characterization shows that the electrical conductivity of both type thin film increases with increasing of BT doping concentration and optimum at concentration of 0.8 wt% for both p-type and n-type thin films, i.e. 17.9 S/cm and 7.78 S/cm, respectively. However, the study of the temperature effect on the thin films electrical conductivity suggested that the thermoelectric properties of both types' samples improved with increasing of BT concentration and optimum at 0.8 and 0.6 wt% for p-type and n-type thin films, respectively. It then decreased if the BT concentration further increased. The Seebeck coefficient for these samples is as high as −11.9 and −15.7 μV/K, which is equivalent to a power factors of 0.26 and 0.19 μS V²/ (m K²), respectively. A thermoelectric device resembling a thermocouple system that was fabricated using the optimum p-type and n-type thin films can generate a voltage as high as 1.1 V at a temperature difference as low as 55 K, which is equivalent to a maximum power of 6.026 μW at V_{max, power} of 0.5489 V (for an estimated matched-load of 50 Ω). The present materials system is potential for powering low power consumption electronic devices.

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1. Introduction

In recent year, electricity power generation using a thermoelectric devices, i.e. a device that generates electricity from a

temperature difference, that work based-on the Seebeck effect is a topic of growing interest [1,2]. This type of thermoelectric device has the advantages of not involving any moving mechanical parts, environmental friendly, less maintenance and easy to control and suited for remote applications for rural power supply [2,3].

The performance of a thermoelectric material under a temperature (T) exposure is determined by three important parameters,

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i.e. Seebeck coefficient (S), electrical conductivity (σ) and thermal conductivity (κ) of the materials [4], which is represented in a Figure of Merit equation of $ZT = (S^2 \sigma / \kappa) T$ [3]. In ideal condition, a thermoelectric material with higher electrical conductivity but with a lower thermal conductivity would ensure the heat at the junctions is retained providing a large temperature gradient [5]. Therefore, to obtain a material with high electrical conductivity but low in thermal conductivity is demanded for an efficient thermoelectric device fabrication.

Bismuth Telluride (Bi_2Te_3) (BT) has been widely studied and commercially developed for a thermoelectric generator applications at 30–100 °C temperature ranges [5]. This material possesses both high electrical conductivity and high Seebeck coefficient [6], which is required for a thermoelectric device fabrication. Unfortunately, BT also exhibits a high-thermal conductivity so that a concrete temperature difference between two points in the material cannot be retained [7]. Therefore, it is required to modify the BT properties, for example via hybridization with organic polymeric materials [8] (such as Poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) (PEDOT:PSS)), to continuously utilize its high thermoelectric properties [9].

The PEDOT:PSS is a highly conductive polymeric material that is widely used as buffer layer in most organic electronic devices. This chemical exhibits excellent optical transparency, chemical stability, and good thermal stability [6,10]. The PEDOT:PSS films have received relatively little attention so far for used as thermoelectric materials because of their lower power factor than that of the state-of-art inorganic thermoelectric materials. Nevertheless, typical thermal conductivity and electrical conductivity of conducting polymers are low [11], but they have the potential to be used as a thermoelectric material by hybridizing them with inorganic semiconductor materials to improve the thermoelectric properties [12]. Such hybrid material may also provide a simpler and cheaper thin film preparation process compared to the conventional inorganic thin film preparation method [7,9].

This paper reports the investigation on the effect of bulk-carrier concentration (BT doping concentration) in the thin films of n-type and p-type BT doped-PEDOT:PSS–Glycerol on its thermoelectrical properties. In typical procedure, the electrical conductivity of both p-type and n-type hybrids films increases with increasing of the BT doping concentration and optimum at 0.8 and 0.6 wt% for p-type and n-type thin films. The electrical conductivity at the optimum condition is as high as 17.9 S/cm and 7.78 S/cm, for p-type and n-type films, respectively. These optimum samples exhibit thermoelectric performance or power factor, namely 0.26 and 0.19 $\mu\text{S V}^2 / (\text{m K}^2)$ for p-type and n-type thin films, respectively. A thermoelectric device fabricated using the optimum condition of both type hybrids films in a thermocouple construction may generate a voltage as high as 1.1 V under a temperature difference as low as 55 K. Owing to the simplicity of the preparation procedure, the BT doped-PEDOT:PSS–Glycerol hybrid film may find a potential use as low-power generator.

2. Experimental

2.1. Materials

PEDOT:PSS solution (PEDOT and PSS content 0.5 and 0.8 wt%, respectively, dispersed in H_2O) and Glycerol 99.5% were purchased from SigmaAldrich. A n-type and p-type of Bi_2Te_3 powder with size of 325 mesh was purchased from Sigma-Aldrich. The chemicals were used as received without any further purification processes.

2.2. Preparation of Bi_2Te_3 doped-PEDOT:PSS–glycerol thin films

Prior to preparing the Bi_2Te_3 (BT) doped-PEDOT:PSS–Glycerol (BT-PPg), PEDOT:PSS–Glycerol solution was firstly prepared by adding 0.1 mL Glycerol directly to 0.9 mL PEDOT:PSS. The solution was then stirred for 12 h at 800 rpm and room-temperature. After that, the solution was filtered through a membrane with pore size of 0.45 μm to remove any traces of un-dissolved aggregates. The effect of Glycerol and PEDOT:PSS concentration on the properties of the p- and n-type hybrid film were also investigated by varying their concentration from 0.05 to 0.2 mL for Glycerol and from 0.8 to 0.95 mL for PEDOT:PSS. The BT-PPg was then prepared by simply adding 0.2–1.0 wt% of p-type Bi_2Te_3 powder to the above solution. This new solution was then stirred for 12 h at 800 rpm and room-temperature. This process was repeated for the preparation of n-type BT-PPg solution.

The thin films of BT-PPg was deposited onto glass substrates by spin coating the solutions at 500 rpm for 30 s. The thin film was then dried on a hot plate at a temperature of 60 °C for 5 min in air. The sample was then transferred into a petri dish and sealed and used directly for further thin films characterization.

For a thermoelectric device fabrication and electrical properties characterization, such as Seebeck coefficient, bulk carrier concentration and electrical conductivity, indium metal contacts were grown at each corner of the substrate containing thin films of BT-PPg by the solder paste technique. The metal was left to dry at room-temperature before further characterization process.

2.3. Thin films characterization

The morphologies and surface roughness of the composite films were observed by a Field Emission Scanning Electron Microscopy (FE-SEM) and an Atomic Force Microscopy (AFM) using a Field Emission Scanning Electron Microscope (FESEM) Hitachi S-4800) operating at an accelerating voltage of 2 kV and the Veeco CP-11 Scanning Probe Microscope system, respectively. The thickness of the composite films was measured by the FE-SEM.

The Hall Effect of the samples was characterized using an ECOPIA HMS-5300 Hall Measurement System with an applied magnetic field as high as of 0.55 ± 0.03 T. The measurement was carried out at room-temperature. The electrical conductivity of the film during the thermoelectric properties characterization was examined using a four-point-probe technique equipped with UNI-T-UT804 Digital Auto Ranging Bench Top Multimeter True RMS I-V measurement system (DC current and voltage resolution of $\pm (0.1\% + 15)$ and $\pm (0.025\% + 5)$, respectively).

2.4. Thermoelectric device fabrication

A thermoelectric device as shown in Fig. 1 was fabricated to acquire the thermoelectric properties of p-type and n-type BT-PPg thin films. A thermoelectric device resembling a thermocouple system was assembled in this study. To make it work as a thermoelectric device, two-set of series p–n junction was realized by shorting the p-type and n-type films with a copper plate. As can be seen from Fig. 1 A, one side of the thermoelectric device was exposed to the heat (hot side) that was controlled by a programmable temperature controller utilizing the UNI-T-UT804 Digital Auto Ranging Bench Top Multimeter True RMS equipped with K-type thermocouple U1183A Air Temperature Probe (resolution $0.75\% \pm 2.2$ °C) and Peltier heater TEC1-04903 (12.7 W). Meanwhile, another side was kept at room-temperature (cold side). Thermoelectric property of the device was examined under temperature difference between the hot and the cold side ranging from 5 to 55 K.

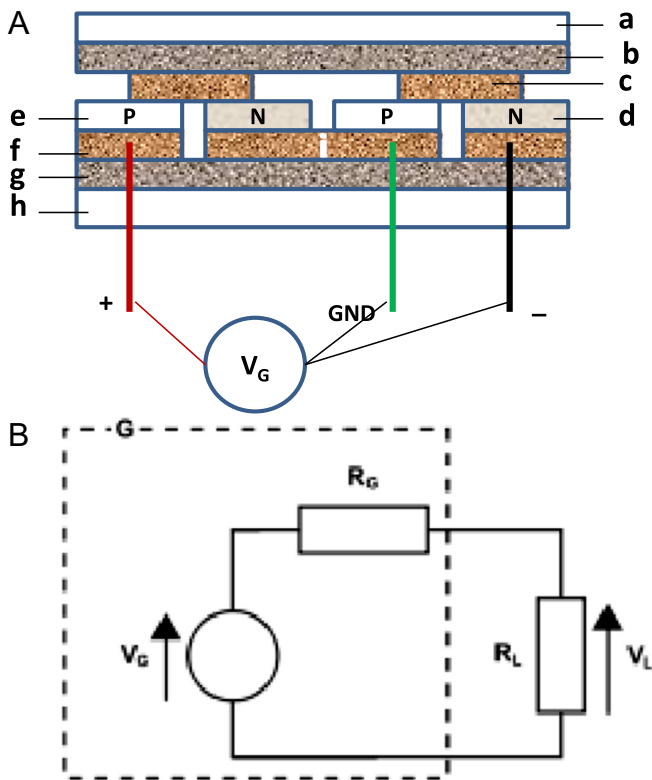


Fig. 1. Block diagram (A) and its equivalent circuit (B) of a thermoelectric device fabricated in this study. The device contains a hot side (a), ceramic insulator (b, g), metal contact (c, f), n-type thin film (d) and p-type thin film (e). V_G and R_G in (B) are open circuit voltage and coupling resistance of the device. Meanwhile R_L is load resistance.

Fig. 1B shows the equivalent circuit for the thermoelectric device. The voltage generated by the thermoelectric device was measured by connecting the p-type and n-type ends altogether with grounding at the center of the structure. The V_G in Fig. 1B is the open circuit voltage (i.e. $i=0$) that is determined by a relation of $V_G = N \times S \times \Delta T$, where N , S , and ΔT are the coupling number, Seebeck coefficient and the temperature difference (ΔT) between the hot and the cold sides, respectively. In this work, the Seebeck coefficient was obtained by interpolating a linear relationship between the voltage gained, V_L ($R_L=50 \Omega$), from the device and ΔT . For the case of the coupling number (N), if two different materials used, the N should be 2 and etc. When the load is attached to the circuit, current will flow through the load and the voltage at the load will be formed. In this situation, the voltage drop on the load was calculated using the relation of $V_L = V_G - iR_G$, where iR_G is a coupling resistant. Maximum power produced from the device is determined by $P_{MAX} = V_G^2 / 4R_G$ at V_{MAX} and I_{MAX} of $0.5V_G$ and $V_G / 2R_G$, respectively. The matched load resistance at the maximum power can be determined by $R_L = R_G (1 + ZT)^{1/2}$, where R_L , R_G and ZT are load, thermoelectric resistance and the figure of merit, respectively.

The figure of merit (ZT) or thermoelectric performance of the material can be calculated by using a relation of $ZT = PF/k \times K$, where PF , k and K are power factor, thermal conductivity of the materials and temperature, respectively. Thermal conductivity (k) of organic polymers is in the range of $0.5\text{--}0.6 \text{ W/(m K)}$. The power factor (PF) for the sample was obtained by using the relation of $PF = S^2 \times \sigma$, where S and σ are Seebeck coefficient and the room-temperature conductivity of the films, respectively.

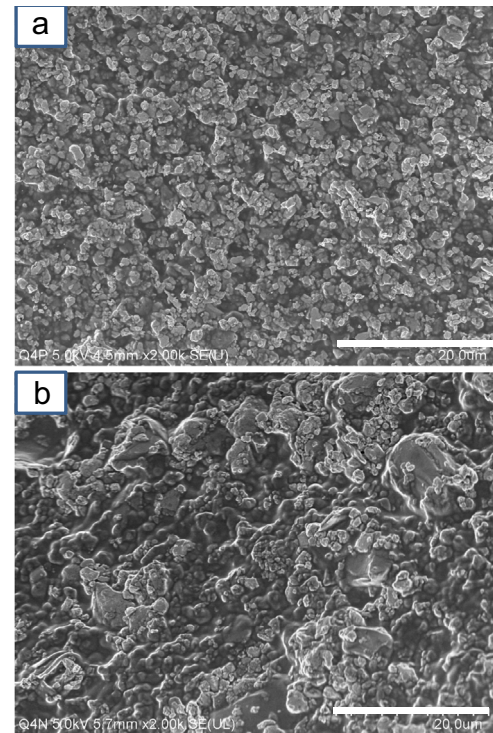


Fig. 2. FESEM image of p-type (a) and n-type (b) BT-doped PEDOT:PSS-Glycerol thin films prepared in the study. The concentration of dopants is approximately 0.8 and 0.6 wt% for p-type and n-type samples, respectively. Scale bars are $20 \mu\text{m}$.

3. Results and discussion

Thin film of p-type and n-type BT-doped PEDOT:PSS-Glycerols (BT-PPg) have been successfully prepared in this work. Fig. 2 shows typical FESEM image of the prepared p- and n-type BT-PPg films with the concentration of dopants of approximately 0.8 and 0.6 wt% for p-type and n-type, respectively. As can be seen from the FESEM results, both thin films samples exhibits that the dopant granules (BT) disperse homogeneously in the PEDOT:PSS-Glycerol matrix, producing highly uniform thin films. Atomic Force Microscopy (AFM) analysis found that the root-mean square (rms) roughness of the film is approximately as low as 487.44 and 540.10 nm for the p-type and n-type film, respectively. This condition is expected to be suitable for fabricating a p–n heterojunction thermoelectric generator device.

Energy-dispersive X-ray (EDX) analysis was then carried out to confirm the type of carrier of the film. The thin films carrier type was determined by the concentration of Bi in the film. The p-type film is obtained if the concentration of Bi is higher than 30% (relative to Te concentration). Otherwise, the thin films is n-type. It was found that the present method has been successfully produced a p-type and n-type BT-PPg thin film. In a typical process, for p-type film, the concentration of Bi is approximately higher than 55%. Meanwhile, the n-type film, the Bi concentration is as low as 10%. The elemental composition of p- and n-type BT-doped PEDOT:PSS-glycerol film are listed in Table 1.

Table 1
Elemental composition in 0.8 wt% of p-type and 0.6 wt% n-type thin films.

| Elements | Symbols | p-type | n-type |
|-----------|---------|--------------|--------|
| | | (atomic wt%) | |
| Tellurium | Te | 33.26 | 25.09 |
| Bismuth | Bi | 32.75 | 4.32 |

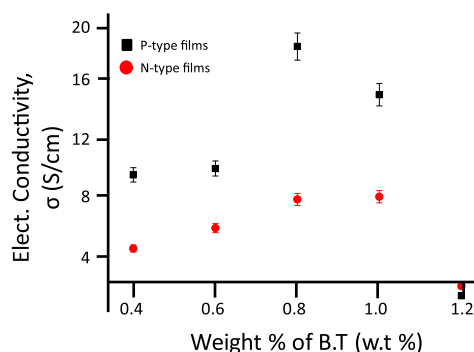


Fig. 3. Electrical conductivity of the p-type and n-type films with various wt% of BT doping at room temperature.

Prior to making the BT-PPg films, the effect of PEDOT:PSS and Glycerol concentration on the thin films properties were evaluated. It was observed that the optimum concentration of Glycerol and PEDOT:PSS for producing high-quality organic films were 0.1 and 0.9 mL, respectively. If the concentrations of both chemicals are beyond this optimum value, the following conditions were obtained. If the Glycerol is higher than 0.1 mL, a good quality film cannot be obtained due to a greasy structure and difficult to dry. Thus, the characterization cannot be carried out. Meanwhile, if the concentration of glycerol lower than 0.1 mL, less-homogeneous thin film with highly cracked structure was obtained. Therefore, this condition will be used for the thermoelectric property of the thin film.

Fig. 3 shows the room-temperature electrical conductivity of the p-type and n-type BT-PPg films with different wt% of BT p-type and n-type. As Fig. 3 reveals, the electrical conductivity of both type thin films increases with the increasing of p-type and n-type concentrations in the thin films. With a coincidence, the electrical conductivities of both type of film are the highest at the concentration of 0.8 wt%. The electrical conductivity of both films then decreases if the doping concentration further increase. On the basis of the FESEM analysis results, such high electrical conductivity in the optimum films, is probably due to the compact structure of the films with a lower surface roughness. It was also observed that the BT dopant distribution as well as its particles size in the films matrix is relatively homogeneous compared to other samples. This could facilitate a facile electrons transport [13]. Thus, increases the electrical conductivity. Meanwhile, in the case of BT doping concentration higher than the optimum condition, an intense dopant aggregation in the film was observed, causing poor electron transport. Thus, increases the resistivity of the films.

While Fig. 3 shows the effect of dopant concentration on the electrical conductivity that was measured at a room-temperature, Fig. 4 presents the electrical conductivity properties of the samples under a temperature variation. As can be seen from Fig. 4a, for the case of p-type film, generally, the conductivity of most of the samples relatively unchanged with the increasing of the temperature. However, for the samples of 0.8 and 0.6 wt%, their electrical conductivity significantly enhanced when the temperature reach 335 K and linearly increase further with the increasing of temperature. At this region, conductivity changes rate as high as approximately $1.57 \text{ S cm}^{-1} \text{ K}^{-1}$ were obtained for both samples. We hypothesized that it is due to the structural-reordering inside the films matrix upon temperature treatment, which generates an exciton Seebeck effect in the molecular system [14]. Enhanced-electron-phonon interplay at high-temperature, especially the in-harmonic character, in the BT system could also be related to the drastic increase in the electrical conductivity if the temperature is increased [15]. These two p-type samples should be potential for thermoelectric device fabrication. Nevertheless, the

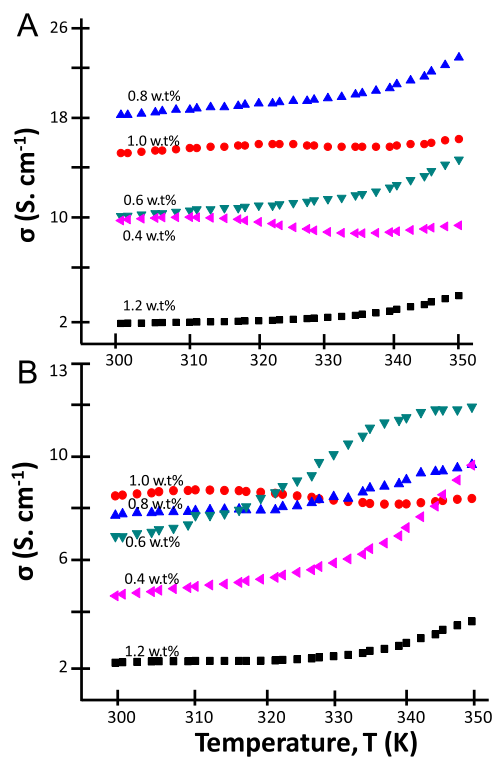


Fig. 4. Electrical conductivity of the p-type (A) and n-type (B) thin films at various temperatures.

0.8 wt% samples show better electrical conductivity than the 0.6 wt% samples, makes it preferred for a thermoelectric device fabrication.

A little bit different behavior is observed in the case n-type film. It was found that the entire samples exhibit significant change in the conductivity when the temperature reached 330 K, which is a little bit lower compared to the p-type film (namely 335 K). Surprisingly, despite room-temperature conductivity of 0.6 wt% samples is lower than the 0.8 wt%, the 0.6 wt% samples shows a lower temperature onset so that enables for a drastic conductivity change, namely at 320 K. At a glance, this reflects that the structural refinement or re-ordering is quickly achieved in the n-type sample so that a further linearly increase in the conductivity with temperature change [13] is obtained. The rate of the conductivity change for sample 0.6 wt% is $0.2 \text{ S cm}^{-1} \text{ K}^{-1}$ compared to $0.063 \text{ S cm}^{-1} \text{ K}^{-1}$ for 0.8 wt%. On the basis of the results, the 0.6 wt% samples is potential for a thermoelectric device fabrication.

We then fabricated the thermoelectric device using the optimum sample of p-type and n-type films. Prior to evaluating the thermoelectric properties of optimum p–n BT-PPg films, individual p- and n-type film's thermoelectric properties were evaluated. In the typical study, two metallic electrodes were grown on the film sample using an evaporation method. One electrode was set as the cold side with temperature controlled at room temperature (i.e. 298 K), meanwhile the other side (hot side) was subjected to temperature change. Fig. 5a shows typical output voltage generated from the p-type BT-PPg (0.8 wt%) upon being applied with a temperature gradient across its two terminals (cold and hot sides). As can be seen from Fig. 5a, the generated voltage linearly increases with increasing of temperature differences and reaches an optimum value at a temperature difference of 55 K (or 353 K at the hot side). At this condition, the voltage generated from the materials is as high as 1.1 mV. Nevertheless, the performance will be dropped when the temperature is further increased from this

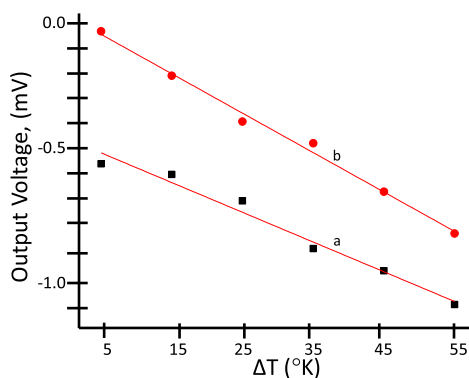


Fig. 5. Performance of thermoelectric device from individual optimum p-type (0.8 wt%) (a) and n-type (0.6 wt%) (b) films.

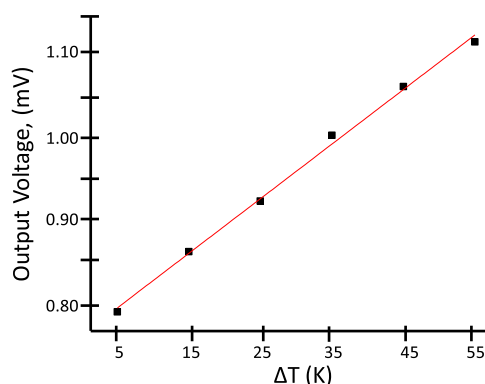


Fig. 6. Performance of a thermoelectric device fabricated using the optimum p-type and n-type thin films with structure shown in Fig. 1 under various temperature differences.

value. This could be simply related to the deterioration or deformation of the thin film structure consisting organic materials that cannot stand at high temperature [11]. Despite the PEDOT:PSS showed relatively limited degradation in the electrical conductivity upon being exposed to relatively high-temperature (i.e. 553 K) [16], its conductivity has been reported to decrease as high as 17% when the material is long-time exposed to a temperature at 380 K [17]. Moreover, the thin film of PEDOT:PSS is susceptible to structural deformation at high temperature, degrading the performance. In addition, although glycerol here is necessary for preparing high-quality hybrids film, high-temperature condition may deteriorate the thin film structure due to its mass lost in the hybrid film structure [18], further adds the limitation to the application at a relatively high-temperature condition. More or less similar properties are also obtained from the n-type BT-PPg samples (0.6 wt%) (see Fig. 5b), in which the voltage generated from the materials is also linearly increase with increasing of temperature difference across the hot and cold side terminal in the materials. For this n-type sample, the optimum temperature difference that gives the best performance is also 55 K. This can be related to the thermal stability of the organic matrix of the film [19], of which is also similar with the p-type one. Nevertheless, the generated voltage from this material is 0.75 mV, which is lower than those produced from the p-type sample.

The Seebeck coefficients (S) for the samples were calculated by obtaining the slope of the curves. It was found that the S values for the p-type and n-type films are -11.9 and -15.7 $\mu\text{V/K}$, respectively. Meanwhile, the power factors of the p-type and n-type films are 0.26 and 0.19 $\mu\text{S V}^2/(\text{m K}^2)$, respectively.

Thermoelectric device was fabricated using the p- and n-type samples with structure shown in Fig. 1. The result is shown in

Fig. 6. As Fig. 6 shows, the output voltage of the device linearly increases with increasing of temperature difference between the cold and the hot side part of the thermoelectric device. From the graph, the Seebeck coefficient of the thermoelectric device is calculated to be as high as 0.06 V/K. This value is comparable to the recently reported result of using BT-organic hybrid film system.

As has been mentioned in the experimental section, the cold side of the device was set at room-temperature, i.e. 25°C . Due to the limited thermal stability of BT-organic hybrids film, the output voltage measurement above the temperature difference of 80° was not examined. It is true that the present device is still low in output voltage, however, owing to its simplicity in fabrication and economically cheap preparation process; the thermoelectric device should find potential application for electrical energy harvesting. High-output power can be simply achieved via multi-plexation used of the device.

4. Conclusion

The effect of BT concentration on the electrical and thermoelectric properties of p-type and n-type BT-PPg film has been studied. It was found that the room-temperature electrical conductivity of both materials (p-type and n-type) increases with the increasing of BT content in the organic films and both are optimum at a BT concentration of 0.8 wt%. The electrical conductivity is as high as 17.9 S/cm and 7.78 S/cm, for p-type and n-type films, respectively. The electrical conductivity decreases when the concentration of BT further increased in the film due to the increase in the structural disorder and surface roughness of the film. In correlation to the electrical conductivity properties, the thermoelectric properties of the p-type and films also indicated an increase with the increasing of BT concentration and optimum at concentration of 0.8 and 0.6 wt% for p-type and n-type, respectively. The Seebeck coefficient of the optimum samples is as high as -11.9 and -15.7 $\mu\text{V/K}$ for p- and n-type films, respectively. Their power factors were found to be as high as 0.26 and 0.19 $\mu\text{S V}^2/(\text{m K}^2)$ for p-type and n-type samples, respectively. A thermoelectric device was fabricated using the optimum samples. It was found that the device can generate a voltage as high as 1.1 V at a temperature difference as low as 55 K, which is equivalent to maximum power of 6.026 μW at $V_{\text{max.power}}$ of 0.5489 V (estimated matched load = $50\ \Omega$). The present system is potential for power generation of low power consumption electronic devices.

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